

METHOD 7041

ANTIMONY (ATOMIC ABSORPTION, FURNACE TECHNIQUE)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000.

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 See Section 3.0 of Method 7000 if interferences are suspected.

3.2 High lead concentration may cause a measurable spectral interference on the 217.6-nm line. If this interference is expected, the secondary wavelength should be employed or Zeeman background correction used.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000.

4.2 Instrument parameters (general):

4.2.1 **Drying time and temp:** 30 sec at 125°C.

4.2.2 **Ashing time and temp:** 30 sec at 800°C.

4.2.3 **Atomizing time and temp:** 10 sec at 2700°C.

4.2.4 **Purge gas:** Argon or nitrogen.

4.2.5 **Wavelength:** 217.6 nm (primary); 231.1 nm (alternate).

4.2.6 **Background correction:** Required.

4.2.7 Other operating parameters should be set as specified by the particular instrument manufacturer.

NOTE: The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20- μ L injection, continuous-flow purge gas, and nonpyrolytic graphite. Smaller sizes of furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above-recommended settings.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 Preparation of standards:

5.2.1 **Stock solution:** Carefully weigh 2.7426 g of antimony potassium tartrate (analytical reagent grade) and dissolve in Type II water. Dilute to 1 liter with Type II water; 1 mL = 1 mg Sb (1,000 mg/L). Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.

5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should contain 0.2% (v/v) HNO₃ and 1-2% (v/v) HCl, prepared using the same types of acid and at the same concentrations as in the sample after processing.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

7.1 Sample preparation: The procedures for preparation of the sample are given in Method 3005. Method 3005, a soft digestion, is presently the only digestion procedure recommended for Sb. It yields better recoveries than either Method 3010 or Method 3050. There is no hard digestion for Sb at this time.

NOTE: The addition of HCl acid to the digestate prevents the furnace analysis of this digestate for many other metals.

7.2 See Method 7000, Paragraph 7.3, Furnace Procedure. The calculation is given in Method 7000, Paragraph 7.4.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

9.2 The performance characteristics for an aqueous sample free of interferences are:

Optimum concentration range: 20-300 ug/L.

Detection limit: 3 ug/L.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 204.2.

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